

producing PVA polymer by saponifying a polyvinyl ester in an alcohol-containing organic solvent under the presence of a saponification catalyst and with which the saponification reaction is carried out while distilling off the carboxylic acid ester that is produced by the saponification reaction.

Claim 55, the now broadest claim, recites:

A method of producing polyvinyl alcohol polymer comprising:

saponification of a polyvinyl ester in an alcohol-containing organic solvent under the presence of a saponification catalyst, wherein saponification is carried out with a mole ratio of alcohol with respect to polyvinyl alcohol of 1.0 to 3.0, while distilling off the carboxylic ester produced by the saponification reaction, wherein said saponification comprises:

a first stage saponification process, comprised in turn of a primary saponification reaction, in which a saponification reaction is carried out in a **kneader mixer** by mixing the polyvinyl ester in the alcohol-containing organic solvent under the presence of a saponification catalyst and

a subsequent secondary saponification reaction, in which a saponification reaction is carried out in a **tower reactor** while distilling off the carboxylic ester that is produced; and

a subsequent second stage saponification process, comprised in turn of a primary saponification reaction, in which a saponification reaction is carried out in a **kneader mixer** by mixing the polyvinyl ester in the alcohol-containing organic solvent under the presence of a saponification catalyst and

a subsequent secondary saponification reaction, in which a saponification reaction is carried out in a **shell and tube evaporator** while distilling off the carboxylic ester that is produced,

wherein the degree of saponification attained in said secondary saponification reaction of the second stage is 99.6 mole% or more and the concentration of the polyvinyl alcohol polymer in the saponification reaction solution is 10 wt% or more.

(Emphasis added.)

As described in the specification under "Description of the Prior Art," beginning at page 1, second paragraph, it is generally believed that in order to raise the degree of saponification of PVA in cases where alcohol is used to saponify a corresponding polyvinyl ester, the mole ratio of methanol with respect to PVA must be made high. However, too high a mole ratio causes other problems, such as PVA gelling, which then requires a large amount of organic solvents, such as dimethyl sulfoxide (DMSO) to prevent gelling, resulting in a concentration of PVA obtained of only a few percent at maximum. Thus, one of the objects of the present invention is a method of producing PVA polymer having a generally higher degree of saponification than that of the prior art, that is obtained at a relatively low mole ratio of alcohol.

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For attaining the above object, as described at page 9, lines 3-21 of the specification, it is preferable to carry out the saponification reaction continuously comprised of a primary reaction, in which a polyvinyl ester is mixed in an alcohol-containing organic solvent under the presence of a saponification catalyst to carry out the saponification reaction, and a secondary reaction, in which the saponification reaction is carried out while distilling off the carboxylic acid ester that is produced.

As described beginning in the paragraph bridging pages 10 and 11 of the specification, and as recited in the present claims, it is further preferable to carry out the above in two stages.

The degree of saponification of PVA can then be increased and the reaction can be controlled by adjusting the mole ratio of alcohol with respect to PVA and the degree of saponification in the secondary saponification can be controlled by adjusting the rate at which the alcohol and carboxylic acid ester are distilled off.

None of the applied prior art addresses the above-discussed problem of the prior art degree of obtaining a relatively high degree of saponification of PVA by using a relatively low mole ratio of alcohol with respect to PVA.

not done

Indeed, such a low mole ratio of alcohol could not possibly be arrived without first coming up with the saponification comprised of the primary and secondary saponification reactions. Note the Examiner's statement at paragraph 11 of the Office Action dated February 4, 2002, referring to Morrison et al and Yanai et al, that in order to shift the equilibrium to the right, it is necessary so far to use a large excess of the alcohol to get PVA polymer which has a higher degree of saponification. Indeed, Morrison et al and Yanai et al actually teach against the present invention. Note the argument above with regard to the disadvantages of too high a mole ratio of alcohol, such as PVA gelling and the consequent need for a large amount of organic solvent.

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The Examiner stated in that Office Action that "[Sato et al] exemplifies hydrolysis reactions in the examples wherein the concentration of the functionalized vinyl acetate polymer is above 10 wt% PVOH. As the hydrolysis reaction has been equated to the ordinary hydrolysis of PVAc, it would have been obvious to one of ordinary skill in the art that such concentration are known to be used in the hydrolysis reactions disclosed for PVAc."

However, the concentration is **not** for PVA but a modified PVA in Sato et al. Sato et al neither discloses nor suggests solving the problem to increase the degree of saponification, and/or to reduce the amount of alcohol required to achieve high degrees of saponification.

In Example 1 in Sato et al, for example, the degree of saponification is 97.0 mol %. But, if the amount of alcohol were reduced, the degree of saponification would become low.

The Examiner stated in that Office Action that shell and tube evaporators are the most widely used type of film evaporator used, among which one version is the falling film type, and in the field of polymer technology it is well known to employ film type evaporators. However, it has been not known to use such a shell and tube evaporator in a saponification reaction, much less to reduce the amount of alcohol required to achieve high degrees of saponification. Indeed, without the present disclosure as a guide, one skilled in the art would not have used a heat-exchanging reactor as recited herein.

As described in the specification beginning in the paragraph bridging pages 12 and 13, in the first stage and second stage saponification processes, it is important for the saponification reactor in which the primary saponification reaction is carried out to reliably mix the saponification reaction stock solution (paste), comprised of polyvinyl carboxylate, alcohol, and organic solvent, and the saponification catalyst, in a short time. From this standpoint, a reactor that is substantially of the complete mixing type is preferably used. Examples of reactors that are close to being such a complete mixing type reactor include line mixers and various other mixers of high mixing efficiency and mixers equipped with various types of stirring blades. Among such mixers, the use of a kneader type mixer is preferable, for example the mixer made under the trade name of S1 Mixer(Type SMJ40) by Sakura Plant Co., Ltd.

As described in the specification beginning in the paragraph bridging pages 14 and 15, if the two-stage saponification, each comprised of first stage, and a second stage saponification processes, is to be employed, a wetted wall tower or tray tower is preferably used for the secondary saponification reaction of the first stage, and a shell and tube evaporator or plate fin type evaporator or an equivalent is preferably used in the secondary saponification reaction of the second stage so as to keep uniform the flow conditions of the PVA polymer solution obtained by the primary saponification reaction. Further, the heat transfer efficiency will be excellent, and methyl acetate can be distilled off readily. Especially in the case where an organic solvent of high boiling point is to be used, the saponification process of the second stage is preferably carried out under highly depressurized conditions, and in order to avoid temperature drops due to sudden flashing, it is preferable to use a shell and tube evaporator, with which the reaction solution obtained by the primary saponification reaction is supplied in an up-flowing manner.

Methyl acetate should be distilled off as much as possible in the first stage in order to achieve a high degree of saponification. Methyl acetate should be 1 wt% or less in the first stage in order to achieve a high final degree of saponification of 99.6 mole % or more. From this point of view, a tower reactor is more advantageous in removing methyl acetate by distillation compared to an evaporator.

In consideration of the above, the best combination of apparatuses is one which can achieve a high degree of saponification of 99.6 mole % or more and a high concentration of the PVA in the saponification reaction solution of 10 wt% or more. The present combination of apparatuses is composed of a kneader mixer as the primary saponification reactor in the first stage, a tower reactor as the secondary saponification reactor in the first stage, a kneader mixer as the primary saponification reactor in the second stage, and a shell and tube

evaporator as the secondary saponification reactor in the second stage, as recited in above-discussed Claim 55.

If, on the other hand, a tower reactor were employed as the secondary saponification reactor in the second stage, a complicated reboiler such as a compulsory circulation type reboiler would be necessary because of the high viscous reaction solution of the secondary saponification reaction of the second stage, which is disadvantageous in equipment cost and operation cost compared to the ordinary thermosiphon type reboiler.

Also, on the other hand, if a shell and tube evaporator were employed as the secondary saponification reactor in the first stage, such a high final degree of saponification as 99.6 mole% or more could not be attained compared to employing a tower reactor, because an evaporator is disadvantageous in distilling off of methyl acetate compared to a tower reactor. A shell and tube evaporator is excellent as a reactor in the secondary saponification reaction in the second stage in order to avoid temperature drops due to sudden flashing by depressurization.

The applied prior art neither discloses nor suggests such a combination of apparatuses in order to achieve a high degree of saponification of 99.6 mole % or more and the concentration of the PVA in the saponification reaction solution of 10 wt% or more.

The newly-submitted Watanabe Declaration demonstrates the superiority of the presently-recited "kneader mixer-tower reactor-kneader mixer-shell and tube evaporator" combination of the present claims, with regard to achieving a final degree of saponification.

In the Watanabe Declaration, Experiment 1 is according to the present invention.

Experiments 2-4 are not. Experiment 2 uses a tower reactor for the second saponification reaction of both the first and second stages; Experiment 3 uses a shell and tube evaporator for the second saponification reaction of the first stage, and a tower reactor for the second

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	MR	DD	
1	IC	IC	
2	T	ST	
3	T	T	
4	ST	ST	
	ST	ST	

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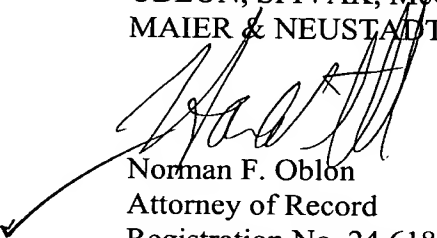
saponification reaction of the second stage; and Experiment 4 uses a shell and tube evaporator for the second saponification reaction of both the first and second stages. A summary of the results is **submitted herewith**. None of the applied prior art could have predicted these results.

For all the above reasons, it is respectfully requested that the rejections over prior art be withdrawn.

All of the presently-pending claims in this application are now believed to be in immediate condition for allowance. Accordingly, the Examiner is respectfully requested to pass this application to issue.

Respectfully submitted,

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IN THE CLAIMS

Claims 29, 31-40, 43-45, 47-50, 52-54 and 59 (canceled)

55. (Amended) A method of producing polyvinyl alcohol polymer comprising:

saponification of a polyvinyl ester in an alcohol-containing organic solvent under the presence of a saponification catalyst, wherein saponification is carried out with a mole ratio of alcohol with respect to polyvinyl alcohol of 1.0 to 3.0, while distilling off the carboxylic ester produced by the saponification reaction, wherein said saponification comprises:

a first stage saponification process, comprised in turn of a primary saponification reaction, in which a saponification reaction is carried out in a kneader mixer by mixing the polyvinyl ester in the alcohol-containing organic solvent under the presence of a saponification catalyst and

a subsequent secondary saponification reaction, in which a saponification reaction is carried out in a tower reactor while distilling off the carboxylic ester that is produced; and

a subsequent second stage saponification process, comprised in turn of a primary saponification reaction, in which a saponification reaction is carried out in a kneader mixer by mixing the polyvinyl ester in the alcohol-containing [organic] organic solvent under the presence of a saponification catalyst and

a subsequent secondary saponification reaction, in which a saponification reaction is carried out in a shell and tube evaporator while distilling off the carboxylic ester that is produced,

wherein the degree of saponification attained in said secondary saponification reaction of the second stage is 99.6 mole% or more and the concentration of the polyvinyl alcohol polymer in the saponification reaction solution is 10 wt% or more.